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# Effect of hafnium on cast microstructure in alloy 1570

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**Abstract:** The issue is devoted to the study of the influence of hafnium on the structure and properties of alloy 1570. Ingots from alloy 1570 were cast into the steel coquille, including those with additives of hafnium 0.1, 0.2 and 0.5 %. To determine the size of the grain structure in the obtained ingots, an Axionovert-40 MAT optical microscope was used, chemical analysis of intermetallic particles was carried out using JEOL 6390A SEM. In addition, for the alloy 1570 and 1570–0.5Hf, the presence of nanoparticles with the $L1_2$ structure was studied using transmission electron microscope JEM-2100. Studies showed that hafnium additives make it possible to achieve a significant modification of the cast structure. For example, when introducing hafnium into the initial alloy in an amount of 0.5 % of the total weight, it was possible to achieve a reduction in the average grain size by 2 times. Scanning microscopy data showed that hafnium partially dissolves in particles containing scandium and zirconium as well. The addition of hafnium increases the number of large particles formed during crystallization. Transmission microscopy showed the presence of coherent aluminum matrix nanoparticles in alloy 1570 and having a superstructure of $L1_2$, which were most likely formed during intermittent decay during ingot cooling. When 0.5 % Hf was added, no nanoparticles with the $L1_2$ superstructure were detected. To explain the latter fact, it is necessary to study the surface of the liquidus of the Al–Hf–Sc system, as well as to study the effect of hafnium on the diffusion coefficient of scandium in aluminum.

**Keywords:** aluminum alloys, alloying with small additives of transition elements, microstructure, intermetallides

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# Влияние гафния на литую микроструктуру в сплаве 1570

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**Аннотация:** Изучено влияние гафния на структуру и свойства сплава 1570. В стальной кокиль были отлиты слитки из сплава 1570, в том числе с добавками гафния (0,1, 0,2 и 0,5 %). Для определения размеров зеренной структуры в полученных слитках применялся оптический микроскоп «Axionovert-40 MAT», химический анализ интерметаллидных частиц проводился с помощью сканирующего электронного микроскопа JEOL 6390A. Кроме того, для сплавов 1570 и 1570–0,5Hf на просвечивающем электронном микроскопе JEM-2100 изучалось наличие наночастиц, имеющих структуру $L1_2$. Исследования показали, что добавки гафния позволяют добиться существенной модификации литой структуры. Например, при введении в исходный сплав 0,5 % Hf (от общей массы) достигнуто уменьшение среднего размера зерна в 2 раза. Согласно данным сканирующей микроскопии, гафний частично растворяется в частицах, содержащих также скандий и цирконий. Добавка гафния увеличивает количество крупных частиц, образующихся при кристаллизации. Просвечивающая микроскопия показала наличие в сплаве 1570 наночастиц, когерентных алюминиевой матрице и имеющих сверхструктуру $L1_2$, которые с большой долей вероятности образовались в ходе прерывистого распада при остывании слитков. При добавке 0,5 % Hf наночастиц, имеющих сверхструктуру $L1_2$, не обнаружено. Для объяснения этого факта необходимы исследования поверхности ликвидуса системы Al–Hf–Sc, а также изучение влияния гафния на коэффициент диффузии скандия в алюминии.

**Ключевые слова:** алюминиевые сплавы, легирование малыми добавками переходных элементов, микроструктура, интерметаллиды

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## Introduction

Aluminum alloys are widely used and largely indispensable in aircraft, rocket and space equipment. Therefore, much research is being done to develop new and improve existing Al alloys [1—6].

One way to improve the performance properties of aluminum alloys is to add small amounts of scandium, the strongest modifier of cast aluminum structure. Furthermore, Sc additions lead to the formation of hardening $Al_3Sc$ nanoparticles when properly cast and heat treated [7].

As a rule, scandium is used together with zirconium, which makes it possible to significantly reduce the amount of Sc required for the cast structure. Moreover, zirconium partially replaces scandium in $Al_3Sc$ particles by forming a shell around them [8], which slows down the coagulation of the particles and increases their thermal stability due to the diffusion coefficient, which is lower for zirconium than for scandium [9].

Aluminum alloys with high magnesium content are the most popular for Sc—Zr alloys because they cause significant solid state hardening [10]. One of the most sought-after alloys of this group is alloy 1570 [6].

Further improvement of the properties of alloy 1570 is possible by the introduction of small additions of hafnium, which, first, is also a strong modifier of the cast structure [11] and, second, like zirconium, partially replaces scandium in the $Al_3Sc$ particles and forms a shell that increases their thermal stability [9]. However, in order to address the question of the relevance of the use of hafnium in this alloy, it is necessary to study its effects on the microstructure of the alloy both in the as-cast state and after heat treatment. Previously, this issue has not been considered anywhere.

The purpose of this study is to investigate the effects of hafnium on the microstructure of alloy 1570 as cast.

## Methods

The samples of the model alloys were prepared under laboratory conditions in the induction furnace UI-25P with an input frequency of 50—60 Hz and an output frequency of 1—20 kHz. The 20×40×400 mm ingots were cast in a water-cooled steel mold at melting temperatures of 720—740 °C. The table shows the chemical composition of all alloys studied.

The following materials were used as furnace charge for the alloy: Aluminum (purity 99.8 %), magnesium (99.9 %), master alloy Al—2%Sc, silumin Al—12%Si, master alloy Al—5%Zr.

The grain structure of the samples was examined using an Axiovert-40 optical microscope MAT (Carl Zeiss, Germany). The average grain size was measured for each sample using the secant method (GOST 21073.2).

Intermetallic particles were examined using a scanning electron microscope SEM JEOL 6390A (Japan). The chemical composition of the structural components was studied by the method of energy dispersive spectroscopy using an X-Max 80T detector (Oxford Instruments, United Kingdom) in the energy range of 0—10 keV (the energy resolution of the detector is 122 eV). The microstructure of the 1570 and 1570—0.5Hf al-

**Investigated alloys chemical composition, %**
Химический состав исследуемых сплавов, %

| Alloy | Al | Si | Fe | Mn | Mg | Ti | Zr | Sc | Hf |
|---|---|---|---|---|---|---|---|---|---|
| 1570 | Basis | 0.17 | 0.27 | 0.44 | 6.16 | 0.03 | 0.05 | 0.22 | – |
| 1570–0.1Hf | Basis | 0.17 | 0.27 | 0.44 | 6.16 | 0.03 | 0.05 | 0.22 | 0.1 |
| 1570–0.2Hf | Basis | 0.17 | 0.27 | 0.44 | 6.16 | 0.03 | 0.05 | 0.22 | 0.2 |
| 1570–0.5Hf | Basis | 0.17 | 0.27 | 0.44 | 6.16 | 0.03 | 0.05 | 0.22 | 0.5 |

loys was further investigated using a JEM-2100 transmission electron microscope (JEOL, Japan) at 200 kV. Elemental analysis was performed using an energy dispersive *X*-ray spectrometer INCA x-sight (Oxford Instruments, UK).

## Results and discussion

Fig. 1 shows the alloy 1570 microstructure with joint scandium-zirconium alloying. This alloy shows equiaxed grain with average size of 44 μm. This is 6 times less than, for example, the high-magnesium alloy 5182 without scandium and zirconium additions [12]. In general, the data obtained correspond to those in [13], where with the joint addition of scandium (0.25 %) and zirconium (0.15 %) to the 1970 alloy, the grain is refined 8.5 times.

With increasing hafnium content, the grain size gradually decreases. With the addition of 0.1 % Hf, the average size of the grain structure decreases to 34 μm.

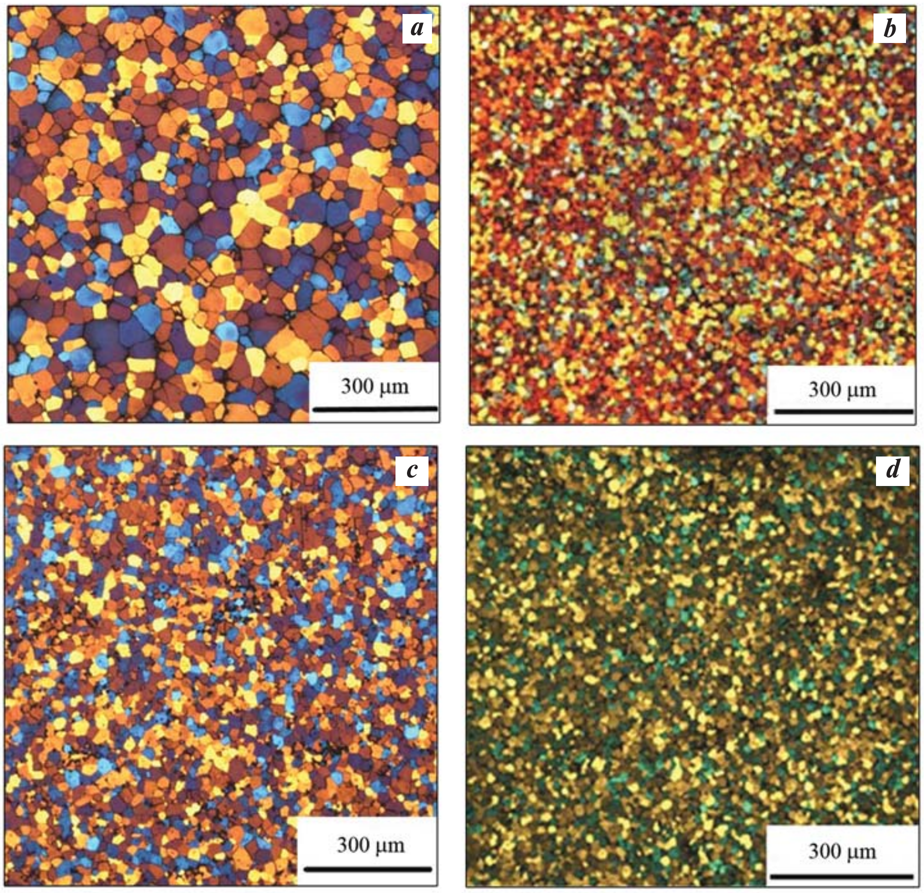


**Fig. 1.** Cast alloy microstructure
***a*** – 1570; ***b*** – 1570–0.1Hf; ***c*** – 1570–0.2Hf; ***d*** – 1570–0.5Hf

**Рис. 1.** Микроструктура литого сплава
***a*** – 1570; ***b*** – 1570–0,1Hf; ***c*** – 1570–0,2Hf; ***d*** – 1570–0,5Hf

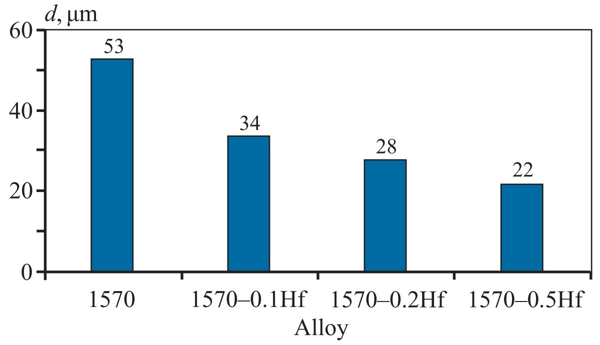


**Fig. 2.** The cast samples average grain size

**Рис. 2.** Средний размер зерна литых образцов

Further increasing the hafnium content to 0.2 and 0.5 % modifies the grain structure to 28 and 22 μm respectively (see Fig. 2).

In order to explain the influence of hafnium on the refinement of the cast grain structure, it should be noted that, according to the Al—Hf state diagram, primary $Al_3Hf$ intermetallides form in the liquid phase at an Hf content of 0.43 % [14]. However, due to non-equilibrium crystallization conditions, primary intermetallic compounds can occur even at an Hf concentration of 0.2 % [11]. In addition, hafnium promotes refinement of the grain structure by reducing the surface tension between the solid and liquid phases, thus facilitating nucleation in the latter [11]. The grain refinement caused by the addition of 0.1 and 0.2 % hafnium can therefore be explained by the reduction in surface tension. A further reduction in grain size with the addition of 0.5 % Hf is due to the appearance of primary $Al_3Hf$ particles.

There may be another explanation for the modifying effect of hafnium when introduced into alloy 1570. Hafnium may have similar effects to zirconium on scandium. Some researchers believe that zirconium reduces the amount of scandium required to reach the proeutectic concentration and initiate the formation of the primary $Al_3Sc$ particles. In this way, according to them, the effectiveness of the joint scandium-zirconium alloy on the change of the cast structure can be explained [15]. The authors of the study [16] show that the common alloying with zirconium and scandium, even at low content of these elements, contributes to the occurrence of primary intermetallic compounds in aluminum alloys. According to the liquidus surface of the Al—Sc—Zr system calculated in [16], the liquid phase starts to crystallize already at low scandium and zirconium concentrations in $Al_{75}Sc_{16}Zr_9$. At scandium and zirconium concentrations typical of alloy 1570, the liquidus surface predicts liquid crystallization into the primary intermetallic phase $Al_2Sc$, which resolves Zr well. Apparently, the occurrence of primary $Al_2Sc$ contributes to the change in grain structure. Hafnium may have a similar effect. However, there are currently no data on the Al—Hf—Sc liquidus surface, making it impossible to confirm this hypothesis.

It should be noted that the EDS analysis can only determine the chemical composition of the intermetallic particles with some accuracy, but unlike the *X*-ray phase analysis, it cannot identify them unambiguously. It is only possible to correlate their chemical composition with the composition of the particles described in the literature, which was done in this study.

Particles containing aluminum, silicon and iron were found in both alloy 1570 and alloy 1570—0.5Hf. In their chemical composition, these intermetallides (*2* and *7* in Fig. 3, *a*, *b*) are close to $Al_3(Fe,Si)$ (Fig. 3, *c*, *d*) [17]. These and similar intermetallic particles are often found in aluminum alloys, since Fe and Si are always present as unavoidable impurities.

The second type of particles found in the study are intermetallic particles containing aluminum and magnesium (*4* in Fig. 3, *b*, *c*). Some intermetallides also contain silicon in addition to the above-mentioned elements (*3* and *6* in Fig. 3). The second type of particles is close in chemical composition to the β-phase ($Al_3Mg_2$), which is very common in high-magnesium aluminum alloys [10, 18, 19], and the third type — the $Mg_2Si$ phase, which is also commonly found in this type of alloys [18, 19]. The presence of aluminum in the particles near $Mg_2Si$ can be explained by the fact that the study with the EDS analysis necessarily captures some of the solid solution.

Another type of particles discovered during the study are particles similar in chemical composition and morphology (diamond-shaped) to the primary $Al_3Sc$ particles (*1* and *5* in Fig. 3) [20, 21]. However, in addition to scandium, they also contain zirconium in alloy 1570 and zirconium and hafnium in alloy 1570—0.5Hf. It should be noted that zirconium and hafnium dissolve in the $Al_3Sc$ phase by 35 and 36 %, respectively [13, 22]. This explains the presence of particles with the combined presence of scandium, zirconium, and hafnium.

When interpreting the transmission microscopy results it should be taken into account that Al has a face-centered cubic unit cell, and $Al_3Sc$ has a primitive cubic unit cell. The primitive lattice will resolve all reflections. According to the energy dispersion microscopy results, we can see the co-directionality of the inverse lattice vectors (<001>, <110>) for $Al_3Sc$ and Al. $Al_3Sc$ and Al also have lattice parameters close to each other, and therefore the resolved reflections for both phases, such as the reflections of surfaces {111},

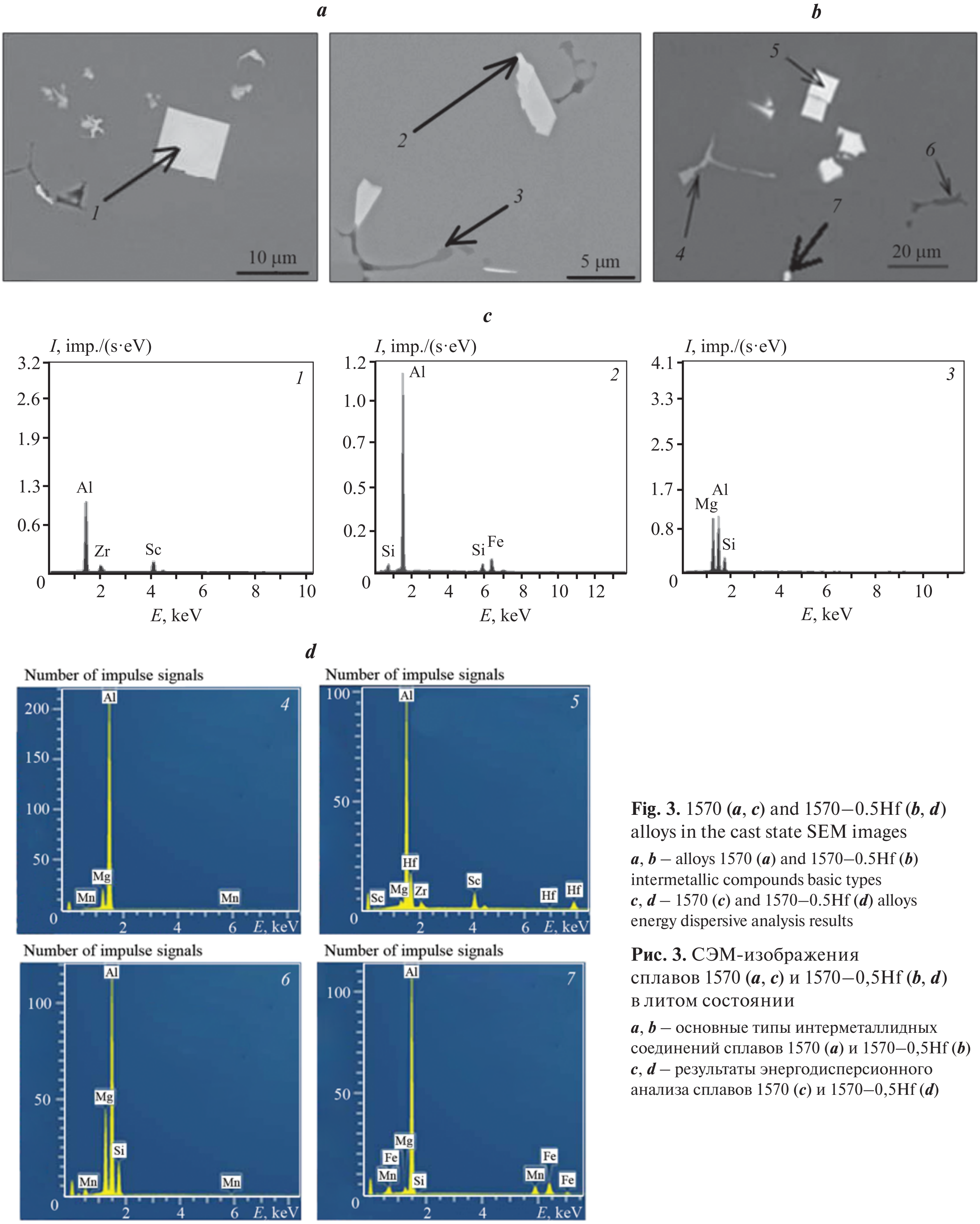


**Fig. 3.** 1570 (***a***, ***c***) and 1570—0.5Hf (***b***, ***d***) alloys in the cast state SEM images

***a***, ***b*** – alloys 1570 (***a***) and 1570–0.5Hf (***b***) intermetallic compounds basic types
***c***, ***d*** – 1570 (***c***) and 1570–0.5Hf (***d***) alloys energy dispersive analysis results

**Рис. 3.** СЭМ-изображения сплавов 1570 (***a***, ***c***) и 1570–0,5Hf (***b***, ***d***) в литом состоянии

***a***, ***b*** – основные типы интерметаллидных соединений сплавов 1570 (***a***) и 1570–0,5Hf (***b***)
***c***, ***d*** – результаты энергодисперсионного анализа сплавов 1570 (***c***) и 1570–0,5Hf (***d***)

agree. Fig. 4, *a* shows three images of microdiffraction in the dark field, where the directions on the zone axis for $Al_3Sc$ and Al coincide. From the above, it can be concluded that the $Al_3Sc$ lattice is coherent with the aluminum matrix. In addition, the presence of {110} type reflexes confirms that $Al_3Sc$ particles have an L12 structure [23, 24]. The average size of the detected nanoparticles was 7—10 nm (Fig. 4, *a*), the results of energy dispersive analysis showed the presence of scandium in them (Fig. 5). As for the 1570—0.5Hf al-

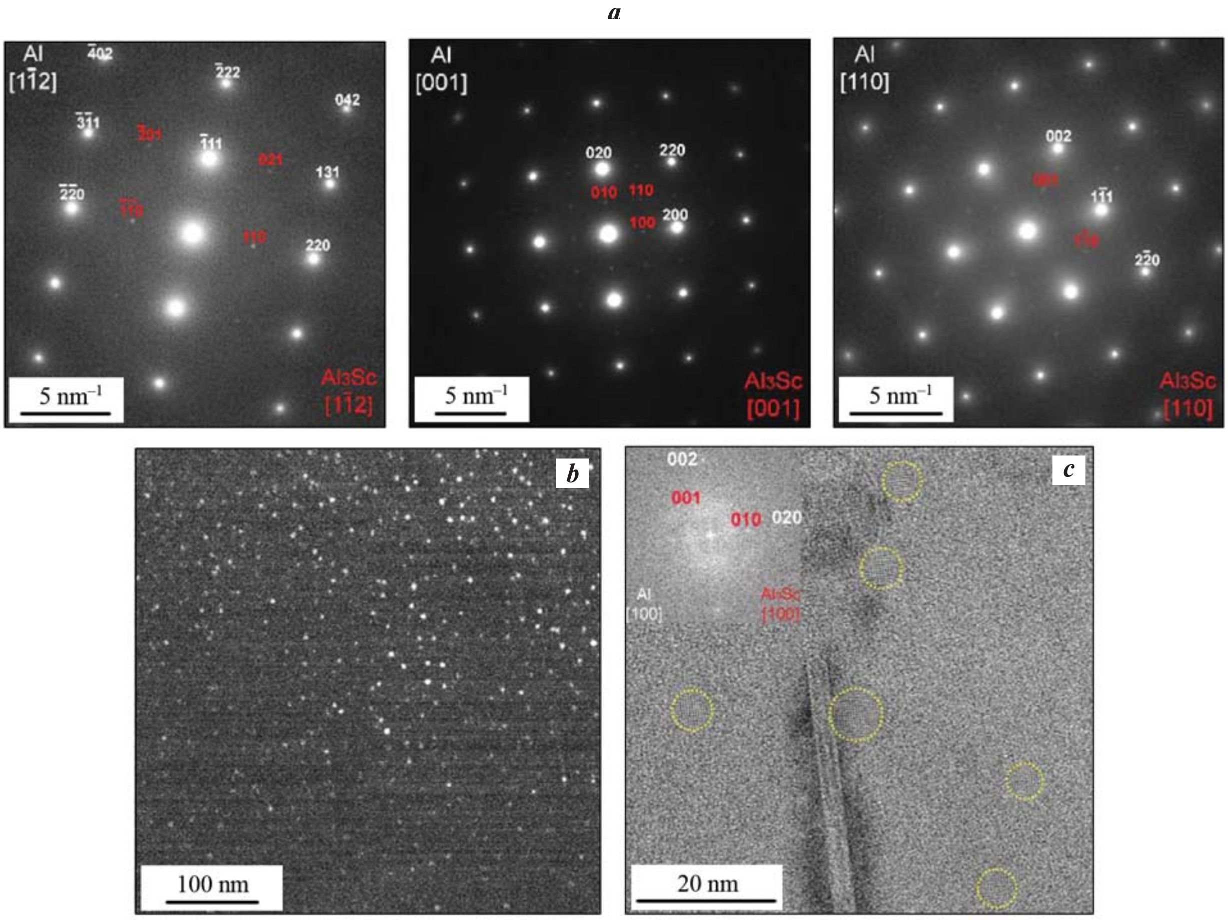


**Fig. 4.** Selected area diffraction pattern of 1570 alloy
***a*** – axes [112], [001] and [110] microelectronograms; ***b***, ***c*** – coherent nanoparticles

**Рис. 4.** Электронно-микроскопические изображения микроструктуры сплава 1570
***a*** – микроэлектронограммы осей [112], [001] и [110]; ***b***, ***c*** – когерентные наночастицы

loy, no particles with reflexes from $L1_2$ superstructures were detected.

Before proceeding to the explanation of the nature and origin of the $Al_3Sc$ particles, it should be noted that when casting aluminum alloys with small additions of scandium, attempts are made to fix the scandium in solid solution due to the rapid cooling of the cast billet in the region of the crystallization temperature [25]. However, due to the high diffusion rate of scandium in aluminum during the subsequent cooling of the ingot, scandium is released in most cases by the intermittent decomposition of the supersaturated solid solution in the form of semi-coherent and coherent particles [20, 21]. Although such particles improve mechanical properties, their efficiency is much lower than that of those produced by continuous decomposition [15]. Moreover, intermittent decomposition of the supersaturated solid solution during cooling of the cast bolt leads to the release of large amounts of scandium, which reduces the number of $Al_3Sc$-type nanoparticles formed during further heat treatment [26]. Therefore, based on the literature data and taking into account the coherence of these particles, the presence of scandium in them and the superstructure $L1_2$, we can say with high probability that they are close to the intermetallic $Al_3Sc$.

The absence of reflections from superstructure $L1_2$ in the alloy with the addition of 0.5 % Hf most likely means that the supersaturated solid solution is significantly lacking in scandium, so that no discontinuous decomposition occurs. One explanation could be the possible effect of hafnium on reducing the proeutectic concentration of scandium mentioned above. When this effect occurs, it means that more primary $Al_3Sc$ particles are formed and thus the scandium concentration in the supersaturated solid solution decreases. This is indirectly confirmed by the increase in the total number of large intermetallic particles (see Fig. 5). Another possible explanation is that hafnium slows the diffusion of scandi-

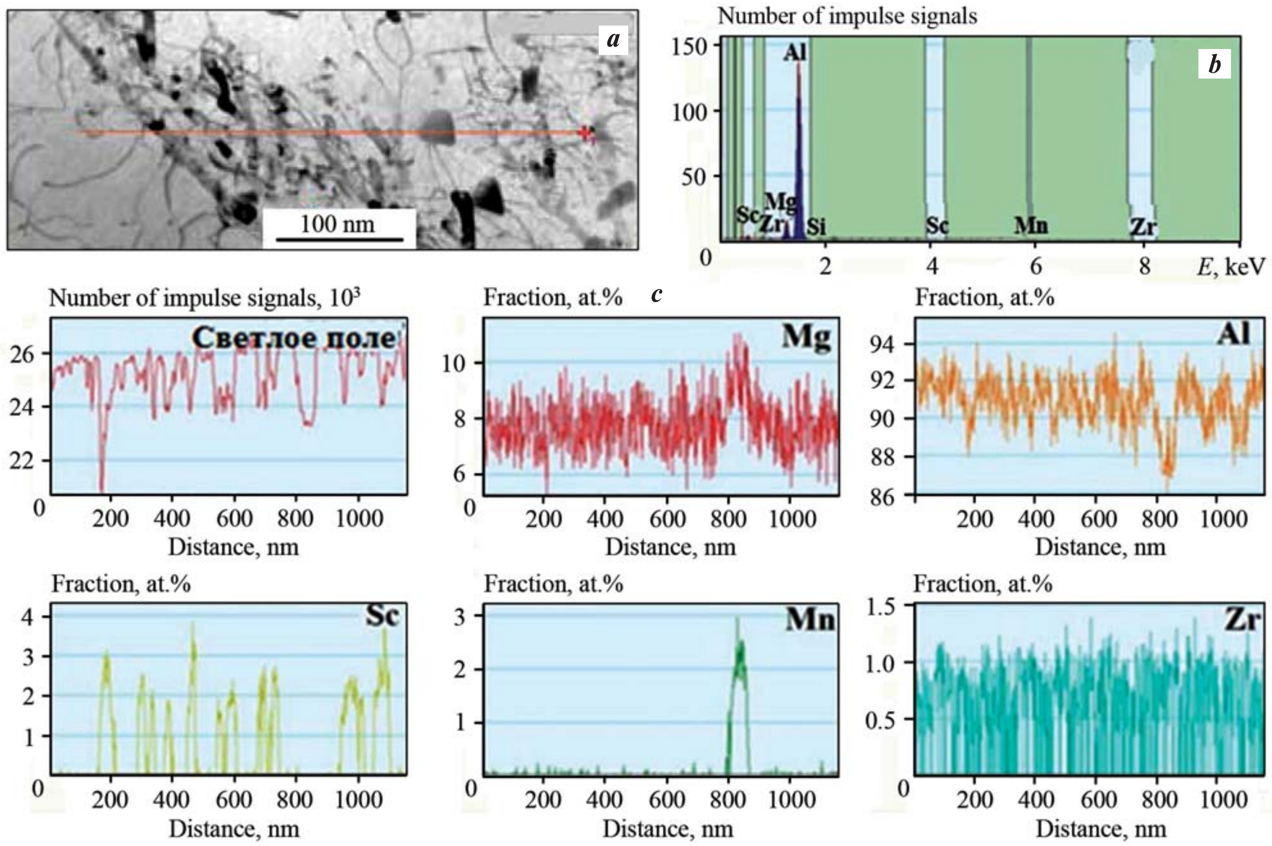


**Fig. 5.** Alloy 1570 energy dispersive *X*-ray spectroscopy

***a*** – investigated microstructure bright-field image; ***b*** – energy dispersive spectral profile (EDS); ***c*** – scanning line (EDS) elements distribution

**Рис. 5.** Энергодисперсионная рентгеновская спектроскопия сплава 1570

***a*** – светлопольное изображение исследуемой микроструктуры; ***b*** – энергодисперсионный спектральный профиль (EDS); ***c*** – распределение элементов по линии сканирования (EDS)

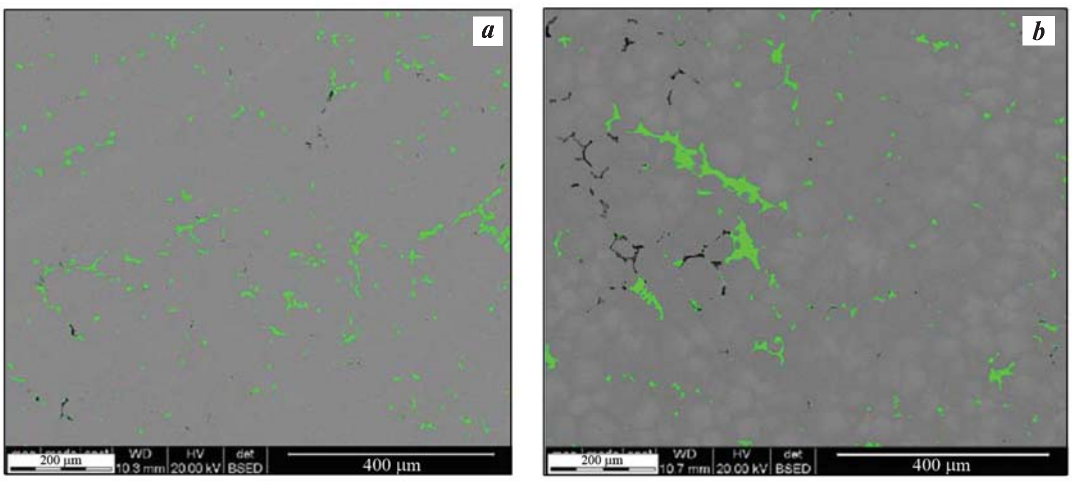


**Fig. 6.** 1570 (***a***) and 1570–0.5Hf (***b***) alloys intermetallic particles appearance

**Рис. 6.** Внешний вид интерметаллидных частиц в сплавах 1570 (***a***) и 1570–0,5Hf (***b***) um in the aluminum matrix, but there are no studies in the literature that address this question.

From the data obtained by scanning electron microscopy (Fig. 6), it can be concluded that the number of intermetallic particles deposited from the supersaturated solid solution increases when alloy 1570 is alloyed with hafnium.

## Conclusion

Hafnium additives allow to increase the efficiency of milling grain by 2 times. The main explanation for this is the primary $Al_3Hf$ particle modifying effect. According to the results of the study the chemical composition of the large intermetallic particles, it was found that hafnium is partially dissolved in intermetallic particles, which also contain zirconium and scandium. Transmission microscopy revealed a large number of coherent scandium-containing particles with superstructure $L1_2$ in alloy 1570, which were most likely formed by intermittent decomposition of the supersaturated solid solution. At addition of 0.5 % hafnium the fine-dispersed particles having $L1_2$ superstructure are absent — to explain this fact additional liquidus surface of Al—Hf—Sc system and hafnium influence on scandium diffusion coefficient investigations are required.

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